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SVE MEMO NO. 125-142

B-5-1

LEADING EDGE & NOSE CAP MATERIALS-PYROLYTIC GRAPHITE

34-P. =3.60

By

H. Morgan

JUNE 30, 1961

Written By:

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Space Vehicle Engineering

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Advanced Methods of Analysis

JUL JIIM 2- 1964

ACKNOWL EDGEMENT

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SUMMARY

A fatigue test of one grade of pyrolytic graphite at room temperature is described bersin, and the resulting preliminary S-N curve is presented. This is the first such data obtained as far as is known.

Also described is a test of pyrolytic graphite hemispheres in the Janus Arc Tunnel, a unique facility which can simulate closely glide re-entry heating. The thermodynamic capability of both the tunnel and the material was thereby demonstrated.

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I. INTRODUCTION

The rapid developments in glide re-entry vehicles will soon reach a point where workable leading edge and nose cap component designs are needed. Structural and thermal requirements which must be met present challenging problems, since temperatures to 5000°F are expected for times approaching one hour. The present status of technology and available materials points to radiative cooling utilizing pyrolytic graphite, in which thermal equilibrium is quickly established between aerodynamic heating and heat loss by radiation as the major mode of heat transfer. Pyrolytic Graphite is a most promising material for these components because of its excellent high temperature strength, good oxidation resistance, and faborable thermal properties (See Reference 1).

Realizing that a need exists for leading edge-type components,

Space Vehicle Engineering (formerly Space Structures Engineering) conducted an Advanced Development Program during 1959-90, reported in document no. TIS RG 61 SD 35. This was a program of sizable scope and dealt with various ramifications of design, fabrication, and testing of a hot load-bearing re-radiative structure. It also included a "Leading Edge Materials Study" in which many ceramic-type materials were screened by exposing button specimens in a plasma flame. The most promising candidate materials were foamed stabilized zirconia, and calcia-compounds.

In addition, a model of a typical pyrolytic graphite leading edge was fabricated and exposed in the Janus Arc Tunnel. This unique

facility had just been completed and was being calibrated at that time. Although the surface eroded severly at the intersection-points of the nozzle shock waves and the model, the test did show capability of both the test facility and the model.

In the six-month period from January 1 to June 30, 1961, a logical continuation of this type of work was planned, consistent with time and funding available, and is described subsequently.

II. DYNAMIC TEST PROGRAM

A. Background

In previous design studies of pyrolytic graphite leading edges and nose caps the dynamic aspects could not be considered, because no dynamic property data of any kind was available. Reviewing all available documents, and questioning many people directly involved in the development of P.G. (pyrolytic graphite), both within and outside the General Electric Company, lead to the conclusion that this information does not exist or has not been made known, and that it is not being obtained. The need for it seems apparent since several semi-operational components are presently being fabricated, and many others are in the design stage (See Reference 2 and 3).

Fatigue problems are the bases of fail-safe structural design which is a characteristic procedure in dealing with a brittle material like. P.G. The absence of yielding creates a number of special problems which must include knowledge of:

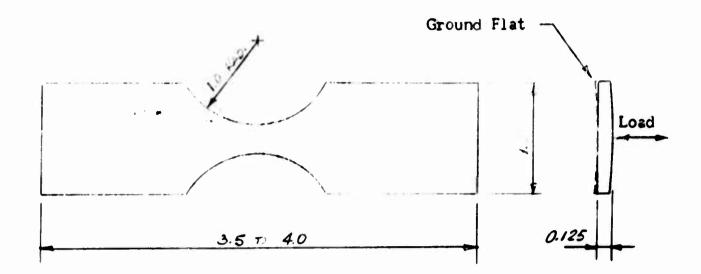
- 1) Accurate static and dynamic loads
- 2) Tolerances
- 3) Material flaws
- 4) Stress concentrations
- 5) Differential expansion

In the design process, it is necessary to know each of these factors, since loads are not redistributed, as they are in a ductile material. The problem is compounded somewhat by the fact that at elevated temperatures, pyrolytic graphite becomes ductile.

It is no small task, of course, to generate sufficient dynamic data for design purposes, as can be seen by examining the broad development program described in Appendix II. The present effort is the first step in this overall program, and satisfies a small portion of an immediate need. The end product is an S-N curve for one grade of pyrolytic graphite at room temperature, and information regarding the mode of failure. A work statement of this program is given in Appendix I.

B. Procedure and Results

- 1. Twenty-seven slabs were cut from a 10 inch I.D. pyrolyticgraphite cylinder of about 0.125 inch wall thickness. Twenty-six were successfully machined into fatigue specimens; one broke in handling.
- 2. To conserve time by simplifying the machining operation, the following specimen shape was accepted, rather than the usual more complicated one indicated in Appendix I:



- 3. Specimens were tested on a Baldwin SF2 machine at 1800 cpm, full reversals.
 - 4. The material density was 2.239 g/cc measured by immersion.
- 5. Figure 1 is a copy of the resulting S-N curve. It must be emphasized that this curve is <u>preliminary only</u>, and must be used with caution and reservation. It is the first such data obtained, and represents only one grade of material at room temperature, and is based on a limited number of test points.
- 6. The average static bend strength was 15,800 psi with a total spread of 3800 psi. Four samples were tested by a one point loading method.
- 7. Five fracture replications and electromicroscope observations were made on specimens with different endurance at equal stress levels, and with similar endurance at different stress levels. No conclusions could be drawn from these limited observations, but with more replications, a reasonable judgment could be formed.

- 8. Enlarged photographs and electronmicrographs were made of the fracture areas of all specimens. A typical set is shown in Figures 2,3, and 4.
- 9. The above work was done at General Electric's Flight Propulsion Laboratory Department, under the supervision of Mr. J. J. Cacciotti.

III. MODEL TEST PROGRAM

A. Background

The final stage of a design is the proof testing of models similar to the final component. For nose caps and leading edges, the testing phase is of major magnitude, since a large amount of heat energy must be impressed on the model for a long time. One such test facility has been developed recently at the Space Sciences Laboratory of MSVD. It is unique in that the new arc heater design permits long time operation at enthalpy values above 10,000 BTU/lb. with contamination by electrode deterioration less than 0.1%. Hence, stagnation point enthalpy of earth re-entry from orbital velocity is closely simulated.

In order to demonstrate the useful application of this tool in evaluating structural components, the testing of several models was planned. In addition to pyrolytic graphite hemispheres, a foamed sirconia leading edge model was fabricated. It was hoped that it could be exposed, thus permitting a qualitative comparison to be made between it and a similar pyrolytic graphite model, mentioned in the Introduction. Due to a series of facility breakdowns, this could not be accomplished in time.

B. Procedure and Results

Four hemispherical pyrolytic graphite specimens were made with the following specifications:

- 1. Diameter: 1.25 in (2 specimens)
 1.00 in (2 specimens)
- 2. Density: 2.1 g/cc
- 3. Wall Thicknesses: 0.250 in.
- 4. Deposition: 200 hr. @ 1700°C; using methane gas
- 5. Cooling: 100°C per hr.

These models were chosen because previous runs indicate that the stagnation point temperature should be 4000°F, which was set as a test requirement to thermally simulate a prototype. Furthermore, a shape of revolution was chosen to avoid thermal complications caused by nozzle shock waves striking the model.

The models were mounted on commercial ATJ graphite stings and held in place by three radial shear pins, also of ATJ graphite. One specimen was mounted on a zirconia sting in an effort to minimize conductive heat loss. Unfortunately, in the machining of this model, the backface surface was pierced, by a drilling operation, to within about 1/16 inch of the front surface, causing a burn through during testing.

One other specimen was exposed with these results:

- Stagnation point temperature-3800° F
- 2. Time-254 sec.
- 3. Surface recession-0.174 in. 9 stagnation point. Material flaked off severely.

The remaining specimens could not be tested due to a generator failure in the arc tunnel power supply, and repairs were made too late to complete the test series.

Based on results of this and other similar tests, there seems little doubt that the target temperature of 4000°F can be sustained for at least 5 - minutes. The use of a low conductivity zirconia sting to avoid heat losses, and a higher grade specimen material to prevent surface spallation, virtually insures this.

IV. Recommendations For Future Activity

There are many aspects of the leading edge and nose cap problem which require major development programs. For example, the feasibility of transpiration-cooling, the use of coated tungsten, or the use of a ceramic. One aspect namely the re-radiative cooling approach using pyrolytic graphite, is detailed in Appendix II. The implementation of this program is most strongly urged, since it would lead to a workable leading edge and nose cap component for glide re-entry vehicles.

REFERENCES

- 1) Morgan, H., "Pyrolytic Graphite Data Summary" SSE Memo 125-122.
- 2) Yaffee, M., "Pyrolytic Graphite Gains As Heat Shields" Aviation
 Week, Feb. 13, 1961 p. 67 ff.
- 3) Yaffee, M., "Pryolytic Graphite Studied For Re-Entry" Aviation Week, July 25, 1960 P. 26 ff.

- TIS RG-61-5D-35

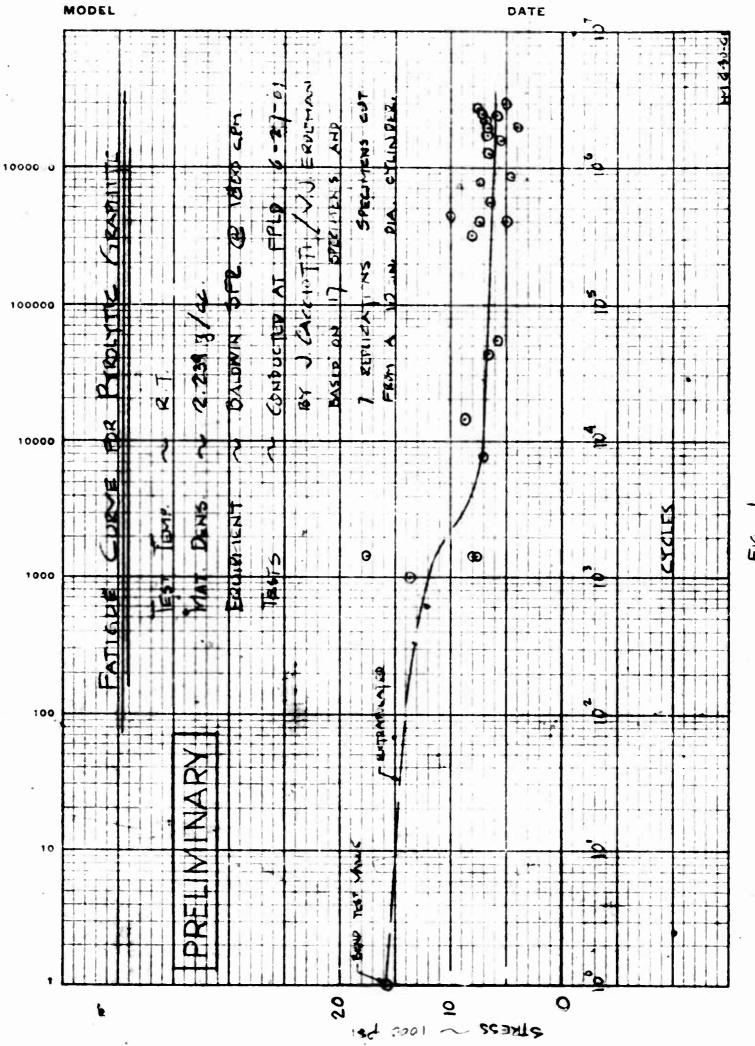


FIG.

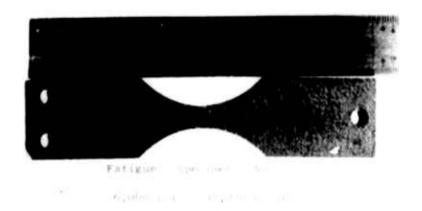


Figure 2. Typical Fatigue Specimen

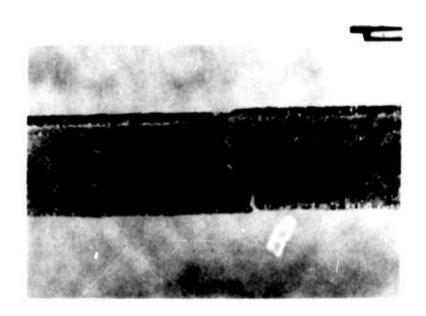


Figure 3. Edge View of Fracture - 17 X

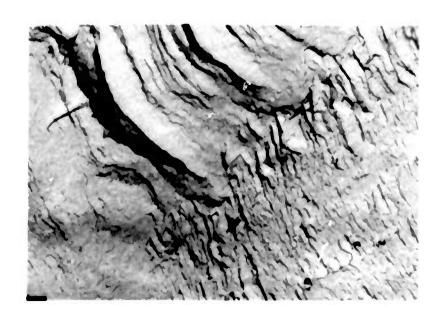


Figure 4. Electronmicrograph of Fracture Face - 7500 X

APPENDIA

APPENDIX I

1) Work Statement Preliminary Engineer

Harry Morgan

2) Number O4-232 Date Space Structures Engineering

- 3) <u>Title:</u> Fatigue Tests of Pyrolytic Graphite
- 4) Deliveries: By sub-contractor to purchaser
 - A. Reports as described in paragraph 6 K
 - B. All specimens, properly identified.
- 5) To Be Negotiated With:

General Electric Company

Flight Propulsion Laboratory Department

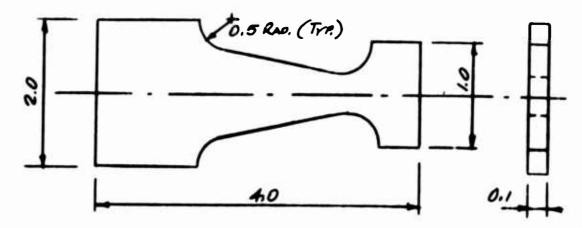
Evandale - Cincinnati 15, Ohio

6) Scope of Work:

- A. In general, the final product desired is a room temperature fatigue curve for one grade of pyrolytic graphite, showing the apparent fiber stress at specimen failure versus the number of stress reversal cycles.
- B. Since this type of work has never been reported in the literature, unforseeable circumstances may require some deviations from the original plan. The subsequent outline attempts to account for this possibility, but leaway must be given.
- C. Fabricate sufficient flat plates for 15 test specimens. The material should have a density of 2.2 grams per cubic centimeter (0.08 lb/cu. in.), be free of delaminations, with a minimum of surface growths.

From these plates cut specimens suitable for testing in a Sonntag

SF-1 vibrator or similar machine, taking care not to marr the surfaces
in any way and should approximate the following shape:



- D) Test two specimens statically to verify the maximum amplitude for failure, (which depends on the specimen used). Load is to be applied perpendicular to the a-b plane.
- E) Using three specimens, more or less, determine the endurance limit as defined by that cyclic stress causing no failure for at least 10 cycles.
- F) For three equally spaced stress levels between the ultimate static and the endurance limit determine the number of cycles to failure, using three specimens for each level.
- G) For each specimen determine whether failure was tensile or shear induced.
- H) If time permits, repeat steps (C) to (G), but this time grind the surfaces down smooth.
- I) Direction of work Jack Graham/ Jos. Cacciotti
- J) Classification U.S. unclassified; GE Co. proprietary

- K) Reports 1) Weekly letter report indicating work completed during that current week and contemplated for the following week.
 - 2) Final report specifying in detail, the methods of fabricating specimens, test procedures, and results.
 Due by June 26, 1961.
- L) <u>Changes</u> Deviations from the work scope as detailed in Section 6 is permissible (see paragraph 6 B), but the essence and aim must be unchanged. In any event, the purchaser and sub-contractor must agree and document any change.
- 7) Specifications: none
- 8) Test and Special Equipment:

Pyrolytic Graphite production furnace

9) <u>Lieison</u>: Refer all technical questions to:

Harry Morgan

Space Structures Engineering

General Electric Company

Missile and Space Vehicle Department

3198 Chestnut Street

Philadelphia 4, Pennsylvania

Work	Statement
Page	4

- 10) Delivery Date: Final Report June 26, 1961
 (Week 26)
- 11) Changes: None (initial issue)

Reviewed By:
A.M. Garber

Prepared By:

H. Morgan, Engineer Space Vehicle Engineering

Approved By:

W. D. McKaig, Manager Space Vehicle Engineering

APPENDIX II

PYROLYTIC GRAPHITE DEVELOPMENT PROGRAM

1.0 INTRODUCTION

1.1 Over-all Problem Roview:

Demands imposed on space vehicle components have been increasing both in severity and complexity, and perhaps the most challenging areas are nose and leading edge portions. Structural requirements of these components present serious problems to the designer, since temperatures of from 3000 to 5000 F, are expected for times approaching one hour. While several heat protection concepts are available such as heat sink, ablation, convection, and transpiration cooling, the most promising on the basis of weight and reliability is radiation cooling. However, it must be recognized that major material and structural problems exist for this type of system. It is necessary to have available materials that possess high strength at extreme elevated temperatures, remain reasonably resistant to oxidation, and also be fabricable.

Pyrolytic Graphite, because of its excellent high temperature strength, good oxidation resistance, and favorable thermal properties, shows great promise as a suitable material for these structural components. Success in utilizing pyrolytic graphite can be achieved only with a detailed knowledge of its behavior when subjected to severe aerothermochemical environment. Its ability to endure the re-entry conditions depends not only upon the rate of application, magnitude, duration, and nature of the heating load, but also upon

its physical, thermal, and mechanical properties which interact with its surroundings. The differing mechanical and thermal properties in perpendicular directions results in behavior which is not similar to that observed in previous aircraft and re-entry vehicle designs.

1.2 Need for Development Program:

The most critical structural problem of the radiative approach deals with finding a high temperature material, and learning how to work with it. This department is already beginning to attain such competency in view of past pyrolytic graphite developments, and the program presented herein is a natural extension of prior work.

Therefore, it is necessary to acquire the ability to design, analyze and fabricate hypothetical nose caps or leading edges, and then demonstrate that these components can be used on various classes of re-entry vehicles.

To do this, a logical Development Program, as presented in subsequent sections, should be followed.

1.3 Problem Areas:

- 1.3.1 Although some basic material property data is available, even to the point of assigning confidence values, additional information is needed, specifically dynamic, creep and oxidation rate data.
- 1.3.2 Pyrolytic Graphite is not new, dating back to 1880 when W. E. Sawyer was granted the first patent in which P.G. seems to be the product, and in 1886 when Thomas Edison produced it in his search for a lamp filament. The process consists of

depositing carbon from a carbonaceous gas onto a mandrel of the required shape. Deposition depends on factors such as gas temperature, flow rate, pressure, composition, etc. Just how these parameters effect the end product quality is not well understood. Hence, some studies must be made to shed light in this area, and guarantee high quality components.

1.3.3 In order to obtain the needed property data, most of which is at elevated temperatures, it is necessary that suitable testing and measurement methods be available. Although this normally belongs in the material properties area, certain test techniques are special problems requiring separate studies. Some of these techniques are known either at MSVE or at GE-sister departments such as FPLD. Others must be developed by means of "sub-development programs."

2.0 OBJECTIVES

2.1 To build prototype nose caps and leading edges which survive in specified glide re-entry vehicle environments. This will be done by first defining types of environments based on typical glide re-entry trajectories. Then, determining necessary material properties and analytical methods which are not available to date. After conducting fabricability and design studies, prototype models will be fabricated and tested.

3.0 PROGRAM DESCRIPTION

3.1 Aerodynamic Environment:

- 3.1.1 Define trajectory classes, i.e., high L/D (lift-to-drag ratio), low L/D, skip glide, and highly maneuverable.
- 3.1.2 Determine for each trajectory, local flow data (temperature, pressure, velocity), which is used in subsequent thermodynamic computations.

3.2 Material Properties:

- 3.2.1 Available data has been surveyed and is presented in Table I and II. They must be reviewed continuously.
- 3.2.2 Static Properties sufficient data is now, or will soon be available for design purposes, with one exception:
 3.2.2.1 Compression yield.

3.2.3 Time Dependent Properties

- 3.2.3.1 Endurance limit use 5 specimens to find the endurance limit and 10 specimens to define a preliminary s-n curve (stress vs. number of loading cycles to fracture To be done at RT, 500°F, 1500°F, 2500°F.
- 3.2.3.2 Damping characteristics use 3 cantilever beam specimens and 2 hinge-ended beam specimens, five readings each at RT, 500°F, 1500°F, 2500°F.
- 3.2.3.3 Notch sensitivity use 3 specimens for each of 3 K factors at RT, 500°F, 1500°F, 2500°F.
- 3.2.3.4 Creep use 36 specimens to obtain stress-rupture curves (stress producing fracture vs. time at that stress) at RT, 500°F, 1500°F, 2500°F (3 points per temperature, 3 runs per point). From these data, plot a Larsen-Miller curve.

3.2.3.5 Oxidation rate - although data is available, it must be verified by testing 2 samples each of 2.2 and 2.1 g/c.c. density pyrolytic graphite, and high density commercial graphite at 1400°, 1800°, 2200°, 2600°, 3600°F.

3.3 Fabrication Studies:

- 3.3₄l Surface growths determine parameters affecting the formation of growths by making about 20 plates, varying for each pressure, temperature, mandrel material. A specific combination schedule cannot be detailed because much depends on information learned from previous runs.
- 3.3.2 Dimensional tolerances; shape limitations, and reporducibility factors make models of four size ranges (subject to furnace availability), three shapes of each size (spherical, cylindrical, and rectangular), three of each (making adjustments every time as indicated). All specimens are to be inspected for size, soundness of material (delaminations, growths), and equivalence of similar models.
- 3.3.3 Machining investigate drilling, threading, and other related problems as they arise.

3.4 Design

3.4.1 Attachments -

- 3.4.1.1 Bonding is being studied under a contract with Lockheed.

 Effort here will consist of keeping up to date with developments.
- 3.4.1.2 Mechanical fasteners clamp, bolt, flange, and breech type joints will be investigated using stress concentration data obtained.

- 3.4.2 Handling, shipping, storage determine factors necessary for the safe handling of components such as container types, handling iigs, and corrosion protection.
- 3.4.3 Thermal shielding due to the high heating rates experienced, provisions must be made to shield the back-up structure and keep temperatures down to tolerable levels.

Alternative methods to be studied are:

- 3.4.3.1 Radiation baffles in which foil gage plates block rearward heat radiation.
- 3.4.3.2 Auxiliary cooling using an active or passive coolant such as water.
- 3.4.3.3 Heat sink providing mass to absorb heat.
- 3.4.3.4 Insulation
- 3.4.4 Sensor provisions investigate the necessity and requirements of temperature, pressure, erosion rate, and stress sensors and provisions for them, such as holes, slots, lugs, etc.
- 3.4.5 Shock loading investigate stresses due to boost, gust, and landing which may be quite high and must be given careful consideration.
- 3.4.6 Acoustic and vibration determine local and over-all stresses due to dynamic effects. Because of the brittleness of pyrolytic graphite, it may become a serious problem, although panel flutter is not expected due to the large wall thicknesses involved.

3.5 Analysis:

3.5.1 Structural

- 3.5.1.1 A survey of methods regarding orthotropic material analysis combined with temperature gradients, to determine what is available and what is needed.
- 3.5.1.2 Extension of available work which presents approximate methods of analysis for orthotropic spherical and cylindrical shells. Include non-circular cross sections, span-wise temperature gradient, variable wall thickness, and variable boundary conditions.
- 3.5.1.3 Residual stresses determine methods of predicting and reducing the effects of residuals.
- 3.5.1.4 Failure determine whether existing yield and fracture criteria are applicable to pyrolytic graphite, and make any modifications necessary.
- 3.5.1.5 Creep and Fatigue analyze the effects of time and cyclic loads, with particular attention to stress concentrations and brittle behavior.

3.6 Models:

- 3.6.1 Design-specimens for property measurements, fabrication studies, detail design studies if necessary, and the prototypes.
- 3.6.2 Fabricate according to results of this and other work.

3.7 Test:

3.7.1 Techniques - develop methods and fixtures necessary for testing of various phases, i.e., property determination, fabricability, and prototype qualification. Tests required are: static, dynamic, thermodynamic (Janus Arc) and pressure-temperature simulation (Malta Rockets).

3.7.2 Measurements - although this is normally included under test techniques, it is an area where a distinct effort is necessary.

For example, measurement of strain at elevated temperature, measurement of residual stress, and orthotropic stress-strain measurement.

23.

4.0 SCHEDULE - see enclosure

ENGINEERING PLAN SCHEDULE PYROCYTIC GRAPHITE DEVELOPMENT PROGRAM

ITEM	MONTHS FROM GO AHEAD										
	0	3	6	9	12	15	18				
AERO ENVIRONMENT		-									
MAT. PROPERTIES		-			\dashv						
FABRICATION		-	-								
DESIGN					\dashv	Į e					
ANALYSIS						-					
MODELS - Properties	-	-	╣.	-							
Fabricability		-		-							
Prototype TEST - Properties				_							
Fabricability		_			-						
Prototype						-					
REPORT											

TABLE I STATUS OF PYROLYTIC GRAPHITE PROPERTIES

			Temperature - OF							
STATIC STRENGTH	*** **** ****	RT	1000	2000	3000	0007	200			
Ultimate Tensile	a b c									
Ultimate Compression	a b c									
Tension Yield	a b c									
Compression Yield	a b c					,				
Ultimate Shear	a b c									
Modulus of Elasticity	a b c									
Modulus of Shear	a b c									
Poisson's Ratio	a b c									
Stress-Strain	a b c									
Modulus of Rupture	a b c									
			,	•	•	1	1			

<u>KEY</u> a - Data presently available

b - Data available by 1962 c - Data needed

TABLE I (cont'd)
STATUS OF PYROLYTIC GRAPHITE PROPERTIES

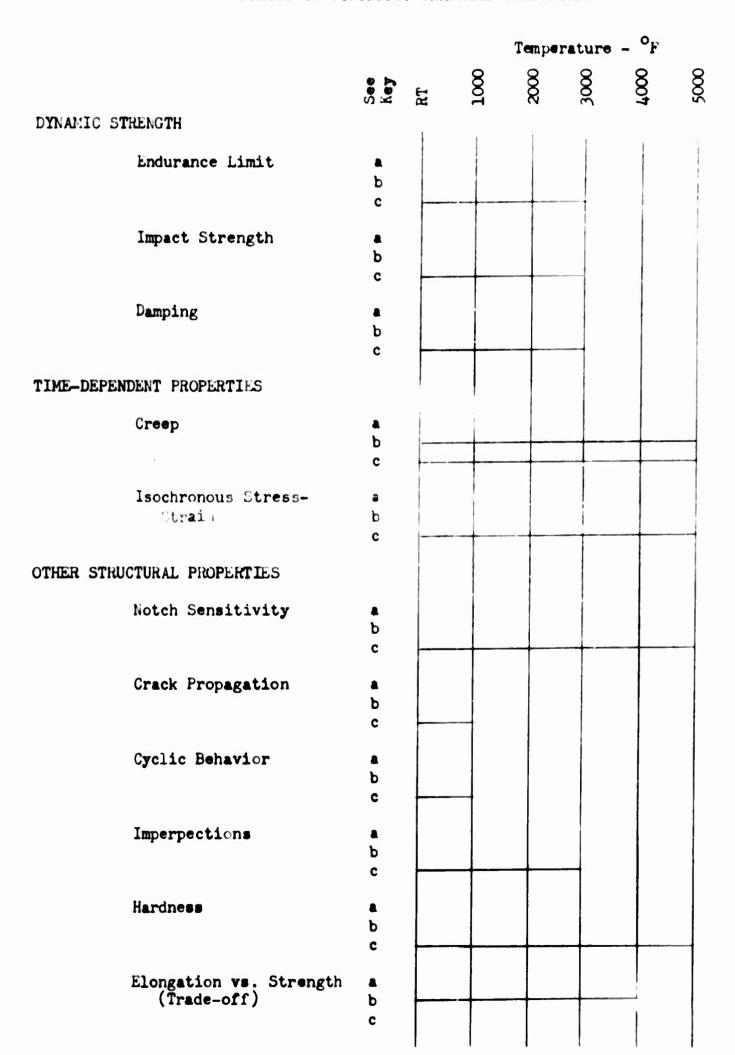


TABLE I (cont'd)
STATUS OF PYROLYTIC GRAPHITE PROPERTIES

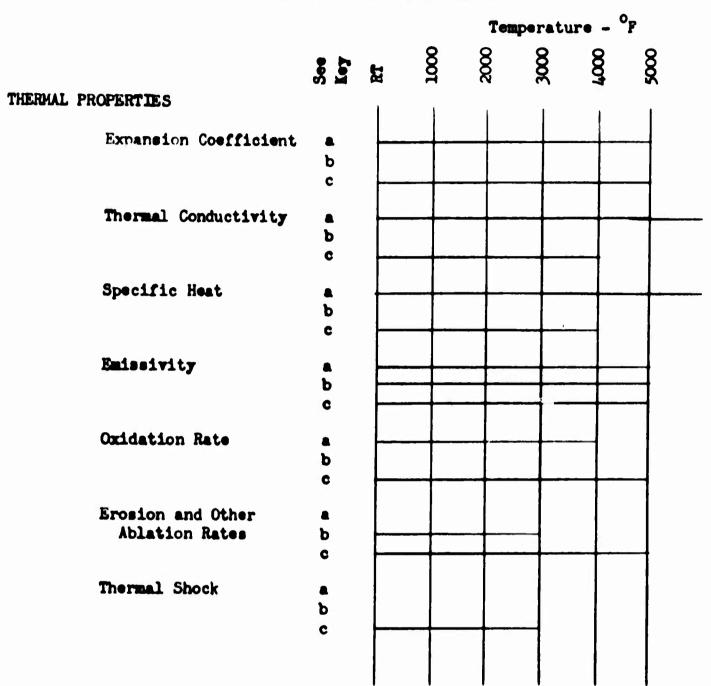


TABLE I (cont'd)
STATUS OF PYROLYTIC GRAPHITE DEVELOPMENT

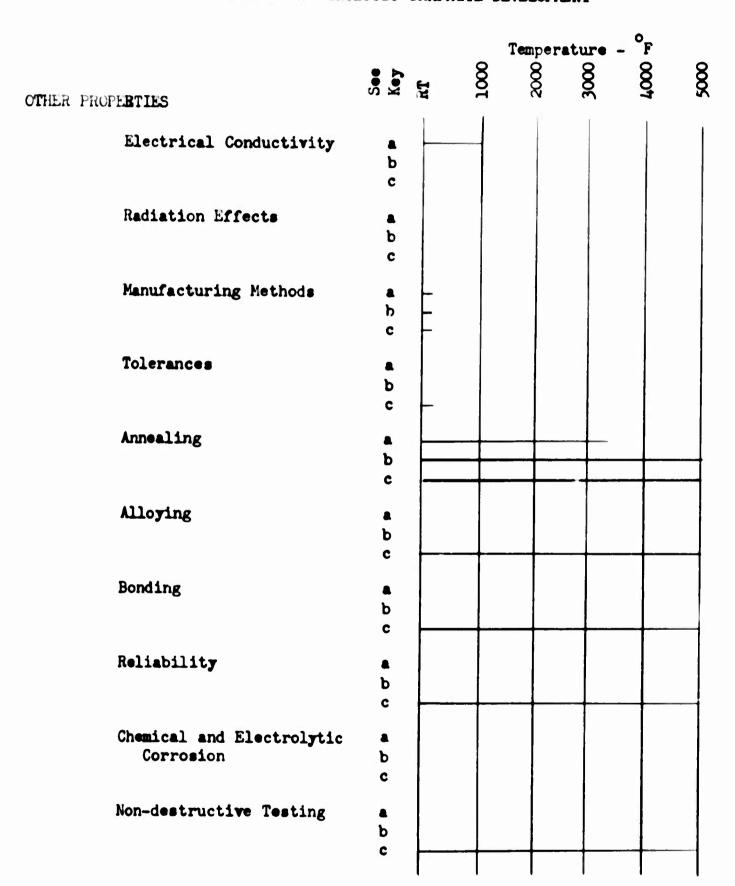


	TABLE II							6) F1					.1				
	PYROLYCIC GRAPHITE	Aerojes - Jeneral	6.	USD - Sunnyrale	11.53 - Palo Alto	78. 78. 78. 78. 78. 78. 78. 78. 78. 78.	ri C	aturataratus com	: 4: Tre 3:	lations] Carbon	34401.6	iening ton Ieos	etals research	Southern	COLLEC	estech.	Chermatest
ī.	STATIC STITEMOTH																
	1. Ultimate Tensile 2. Ultimate Compression 5. Tension Yield 4. Compression Yield 5. Ultimate Shear 6. Mod. of Elesticity 7. Mod. of Shear 8. Poisson's Ratio 9. Stress-Strain 10. Modulus of Rupture	ж.	x x x x x	x x	×				x x x x								x
II.	DYMAMIC STREATTH																
	 Endurance Limit Impact Strength Damping Characteristics 						х										
III.	TIME-DEPENDENT PROP.																
	1. Creep 2. Isochronous Stress-Strain	×	X		×												×
IV.	1. Notch Sensitivity 2. Crack Propagation 3. Cyclic Behavior 4. Laperfections 5. Elongation vs. Strength 6. Hardness 7. Density		x	×	×		x	×								-	
٧.	THEREAL PROPERTIES															1	
	1. Coefficient of Expansion 2. Thermal Conductivity 3. Specific Heat 4. Emissivity 5. Oxidation Fates 6. Prosion and Ablation 7. Thermal Shock 8. Hot Flow Characteristics				x x	81	x x x	×	×	x x	×	x	x x x	x x x			
VI.	OPH 21																
	1. Electrical Conductivity 2. Madiatio. Diffects 3. Manufacturing Methods 4. Polerances 5. Annealing 6. Alloying 7. Donding 6. Miloying 7. Donding 7. Donding 8. Miloying 9. Chem. and Mecurolytic Con 10. Don-destructive Testing 11. Detallography 12. Ultrasonics 13. Addy Current 14. Dye Penetrant 15. Pariography 16. Societ	n.		* * * * * * * * * * * * * * * * * * *	x x	x	* * * * *	* * * * * * * * * * * * * * * * * * *		x .							
	17. X-ray Diffraction = 18. Brazing 19. Coating			x	x x x			X									
	20. Permeability				×												

Avco

TABLE II (continued)

A. Agencies extensively engaged in work with P.G.:

- 1. Jet Propulsion Laboratory
- 2. LMSD Sunnyvale
- 3. LMSD Palo Alto
- 4. LMSD Van Nuys
- 5. G. E.
- 6. High Temperature
- 7. Raytheon

B. Agencies moderately engaged in work with P.G.:

- 1. National Carbon
- 2. Battelle
- 3. Lexington Labs
- 4. Metals Research
- 5. Southern Research
- 6. Narmco
- 7. Westech
- 8. Solar
- 9. Avco
- 10. Aerojet-General